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(54) [Title of invention] Production Method of Polyester with High Degree of Polymerization

(57) [Abstract]

[Issues]

A production method to obtain polyester with a high degree of polymerization in an open ring polymerization using cyclic polyester oligomers in a shorter time period compared to that with a prior art method.

[Means of Resolution]

A production method of polyester with a high degree of polymerization in which, in an open ring polymerization of cyclic polyester oligomers, the cyclic polyester oligomers are blended with polyester with a weight ratio of 0.01 ~ 0.9 against the polyester, then heated at a temperature in a range of 200 ~ 320 degrees Celsius, and thereby polyester with a high degree of polymerization is obtained.

[What is claimed is:]

[Claim 1]

A production method of polyester with a high degree of polymerization, wherein when cyclic polyester oligomers and polyester whose intrinsic viscosity is 0.5 ~ 1.5 are blended and heated to obtain polyester having an intrinsic viscosity of 0.65 or more, the cyclic polyester oligomers are blended in a range of 0.01 ~ 0.9 in weight ratio against said the polyester, and a temperature at the time of heating is in a range of 200 ~ 320 degrees Celsius.

[Claim 2]

The production method of polyester with a high degree of polymerization according to claim 1, wherein at the time of blending and heating, a compound which includes at least one selected from a group comprising of Ti, Sb, Bi, Sn, Al, Ge, Zr, Hf, Y, Sc and lanthanoid compounds (La, Ce, Sm, Eu, Yb) is used as a catalyst.

[Claim 3]

The production method of polyester with a high degree of polymerization according to claim 1 or claim 2, wherein when the cyclic polyester oligomers, the polyester and the catalysts are blended, an extruding machine is used.

[Claim 4]

The production method of polyester with a high degree of polymerization according to claim 3, wherein the extruding machine is a 2-axis extruding machine.

[Claim 5]

The production method of polyester with a high degree of polymerization according to claim 3, wherein the extruding machine is a 1-axis extruding machine with vent or 2-axis extruding machine with vent.

[Detailed Explanation of the Invention]

[0001]

[Technical Field of Invention]

The present invention is related to an open ring polymerization of cyclic polyester oligomers. To be more specifically, the present invention pertains to a production method in which the ratio of cyclic polyester oligomers and polyester is set appropriately and thereby polyester with a high degree of polymerization is obtained in a short time period.

[0002]

[Prior Art Technologies]

Because of its excellent properties, polyester is used for a wide variety of areas such as for fibers, films and bottles. In particular, polyalkylene terephthalate is superior in its mechanical strength, chemical properties, dimensional stability and so forth, and thus it is preferably used. Besides, among its uses, for example, for fibers to be used in industrial materials, a high degree of strength is required. Therefore, methods to improve the fiber strength through making the molecular weight of polyalkylene terephthalate higher is suggested.

[0003]

Generally, polyalkylene terephthalate is produced from terephthalic acid or its ester formation derivative and alkylene glycol, but in a commercial process to manufacture polymers with a high molecular weight, a method in which a melt polymerization is conducted and then a solid phase polymerization is conducted to obtain a high degree of polymerization is widely applied. However, a method to obtain a high degree of polymerization through a solid phase polymerization has some undesirable characteristics such as those which are described below.

[0004]

For instance, in order to obtain polyethylene terephthalate with a high degree of polymerization, a production is conducted using terephthalic acid or its ester formation derivative and ethylene glycol. Since they are advantageous in terms of costs, a direct polymerization method using terephthalic acid, and further a continuous polymerization method are applied. Moreover, in order to obtain even higher polymerization degree, a method with a solid phase polymerization is known. Advantages of the solid phase polymerization method include that it can control oligomers and acetaldehyde that are contained in polymers. However, in order to conduct a solid phase polymerization, there is a large increase in the cost for facilities such as a process for drying polyester chips. Therefore, a significant improvement of its productivity is desired.

[0005]

For these issues, for instance in publication No. WO96/ 22319, a method is proposed in which polyethylene terephthalate prepolymer in a molten state having a polymerization degree of 5 ~ 35 is cooled at 120 ~ 210 degrees Celsius, then pelletized and crystallized at the same time, or in which non-quality polyethylene terephthalate prepolymer in a pellet form having a polymerization degree of 5 ~ 35 is rapidly heated to 120 ~ 210 degrees Celsius and then crystallized, and a solid phase polymerization is conducted at 230 ~ 240 degrees Celsius to give polyethylene terephthalate prepolymer. With these methods, a melt polycondensation process is eliminated and a solid phase polymerization is conducted. Thereby it proposes a reduction of facility costs. However, in order to obtain a higher polymerization degree with polyethylene terephthalate, solid phase polymerization facilities and a substantial reaction time is required. Therefore, these methods are still insufficient in view of the productivity.

[0006]

On the other hand, in recent years open ring polymerization reactions using cyclic polyester oligomers are attracting attentions. In Japanese Laid Open Patent Publications Nos. H8-53474, H8-225573 and H9-3178, an open ring polymerization of cyclic polyester oligomers is reported. It is reported that heating cyclic polyester oligomers allows polymers with a high degree of polymerization to be obtained in a very short time period. However, the melting point of cyclic polyester oligomers is higher than that of straight polyester oligomers. Therefore, higher temperatures are used to melt polyester oligomers than in prior art polyester production methods

[0007]

In this connection, in order to solve the aforementioned issues, in Macromolecules (Robert R. Burch, 33 (5053), 2000), it is described that cyclic polyethylene oligomers and polyester are blended and heated with a weight ratio of 50 : 50 or 1.0, and then an open ring polymerization is conducted at a temperature which is lower than the meting point. In the report, the polymers are used together, and thus the reaction proceeds at 280 degrees Celsius, which is a lower temperature compared to an open ring polymerization with cyclic trimers alone. However, polyester having a sufficient strength cannot be obtained.

[0008]

Furthermore, Japanese Laid Open Patent Publications No. H6-228291 exhibits a provision of shaped products of polyester whose starting material is cyclic polyester oligomers and that are superior in bending strength and are crack-proof. In an open polymerization of cyclic polyester oligomers, in order to reduce the melting heat for said composition to 60J/g or less, polyester in a cyclic or straight form are used together with a mole percentage of 8 ~ 30 against cyclic polyester oligomers. In an example, heating was conducted at 190 degrees Celsius for 10 minutes to obtain polyester. However,

in the publication, polyester with a high degree of polymerization and strength cannot be obtained simply by blending and heating cyclic polyester oligomers and polyester. In addition, it is also reported in the publication that when straight polyester is used in a mixture for cyclic polyester oligomers, the viscosity of cyclic polyester oligomers increases wrongly and thus it is not preferable.

[0009]

Based on the background described in the above, it is desired to economically obtain polyester with a high degree of polymerization.

[0010]

[Issues that the Invention Attempts to Solve]

The purpose of the present invention is to eliminate the aforementioned disadvantages in a production method of polyester with a high degree of polymerization, and to provide a production method of polyester with a high degree of polymerization using an open ring polymerization.

[0011]

[Means to Solve Issues]

The aforementioned purpose of the present invention is accomplished by a production method in which when cyclic polyester oligomers and polyester having an intrinsic viscosity of 0.5 ~ 1.5 are blended and heated to obtain polyester with an intrinsic viscosity of 0.65 or more, the cyclic polyester oligomers are blended in a range of 0.01 ~ 0.9 in weight ratio against said polyester, and heated at a temperature in a range of 200 ~ 320 degrees Celsius.

[0012]

[Embodiments of the Invention]

Polyester of the present invention is not particularly limited as long as it is a polymer that is synthesized from a dicarboxylic acid or its ester formation derivative, and diol or its ester formation derivative, and as long as it can be used as shaped products such as fibers, films, bottles and the like.

[0013]

As concrete examples of such polyester, polyethylene terephthalate, polypropylene terephthalate, polybutylene terephthalate, polycyclohexylene dimethylene terephthalate, polyethylene-2, 6-naphthalene dicarboxylate, polyethylene-1, 2-bis (2-chlorophenoxy) ethane-4, 4'-dicarboxylate and the like can be cited. The present invention is especially suitable with polyethylene terephthalate, polypropylene terephthalate and polybutylene terephthalate that are generally used, or with polyester copolymers which mainly contain polyethylene terephthalate, polypropylene terephthalate and polybutylene terephthalate.

[0014]

Furthermore, to such polyester, as copolymerization components, dicarboxylic acids and their ester formation derivatives such as adipic acid, isophthalic acid, sebacic acid, phthalic acid, and 4, 4'-diphenyl dicarboxylic acid, dioxy compounds such as polyethylene glycol, diethylene glycol, hexamethylene glycol, neopentyl glycol and polypropylene glycol, oxycarbonic acids and their ester formation derivatives including p-(β -oxyethoxy) benzoic acid and lactic acid may be copolymerized.

[0015]

In the present invention, cyclic polyester oligomers are made from polyester materials shown in the above. Regarding the production method, prior art methods can be employed and it is not particularly limited. For example, in Polymer Letters (F. Lynn Hamb, 5 (1057), 1967), a synthesizing method of cyclic polyester oligomers is reported in that 1-methylnaphthalene is used as a solvent, and tetraisopropyl titanate is used as a cyclization catalyst. Furthermore, Japanese Laid Open Patent Application Publication H8-225633 suggests to employ a hydrocarbon such as ortho-terphenyl, meta-terphenyl or para-terphenyl and the like as a solvent, and an organic tin compound and/ or a tetraalkoxy titanate including tetrabutyl titanate as a cyclization catalyst. Therefore, as a solvent to be used in the present invention, a hydrocarbon including 1-methylnaphthalene, ortho-terphenyl, meta-terphenyl or para-terphenyl and the like can be used, for a cyclization catalyst, an organic tin compound and/ or tetraalkoxy titanate and so forth can be employed, but it is not particularly limited. In addition, it is preferred that the amount of cyclization catalysts to be added is 100 ~ 20000ppm in metal weight against cyclic polyester oligomers to be obtained considering a reaction speed of a cyclization reaction and an economical efficiency, and 1000 ~ 10000ppm is more desirable.

[0016]

In the production method of polyester with a high degree of polymerization, first, when blending and heating cyclic polyester oligomers (A) and polyester (B), it is necessary to conduct the blending with the weight ratio A/B of 0.01 ~ 0.9. The ratio of 0.2 ~ 0.7 is preferable and that of 0.3 ~ 0.5 is more desirable. When the weight ratio A/B is less than 0.01, the number of cyclic oligomers to connect rings of polyester is small, and thus polyester with a high degree of polymerization can not be obtained. On the other hand, when the weight ratio A/B exceeds 0.9, a substantial reaction time is needed to open rings of cyclic oligomers to be reacted, and thus it is not desirable. Moreover, in the present invention, the intrinsic viscosity of polyester that is blended with cyclic polyester oligomers is 0.50 ~ 1.50, preferably 0.55 ~ 1.00, and 0.65 ~ 0.85 is even more desirable. Besides, polyester to be used in the present invention can be produced with a prior art method, and it is not particularly limited.

[0017]

In the production method of polyester with a high degree of polymerization of the present invention, cyclic polyester oligomers and polyester should be heated at temperatures in the range of 200 ~ 320 degrees Celsius. At this point, if the temperature is lower than 200 degrees Celsius, it is difficult to melt a mixture of cyclic polyester oligomers and polyester, and after melting, a reaction speed is slow. Therefore, it is not preferable in view of an economical efficiency. On the other hand, when the temperature is higher than 320 degrees Celsius, the mixture is easily melted. However, it is not desirable considering a degradation of the polymer quality which is caused by thermal decomposition of generated polyester. Therefore, the temperature is preferred to be 220 ~ 310 degrees Celsius, and 240 ~ 300 degrees Celsius is more preferable. In addition, regarding the heating method,

heating can be conducted at a fixed temperature. In order to obtain polyester with even higher degrees of polymerization, a temperature control including increase and decrease of temperatures can be conducted.

[0018]

For polymerization catalysts to be used in the present invention, compounds that contain at least one selected from Ti, Sb, Bi, Sn, Al, Ge, Zr, Hf, Y, Sc and lanthanoid compound (La, Ce, Sm, Eu, Yb) are desirable. It is more preferred to use compounds having Ti, Sb, Bi, Sn and Al. As polymerization catalysts, one or more of the compounds mentioned in the above can be used, and a method to add multiple compounds or a method to add compounds made of a plurality of metals can be applied.

[0019]

Concrete examples of tin compounds include, dibutyl tin oxide, stannous chloride, tin octylate, tin laurilate and monobutyl hydroxy tin oxide and so forth. These tin compounds can include those in which a plurality of tin compound molecules are associated or those which became oligomers with some changes.

[0020]

Moreover, as examples of titanium compounds, titanium alcoxide compounds such as tetrabutyl titanate and tetraisopropyl titanate, titanium compounds containing chelating agents such as ethylenediamine tetraacetic acid, hydroxy ethyl iminodiacetic acid, diethylenetriamine pentaacetic acid, triethylene tetramine hexaacetic acid, citric acid, maleic acid or a mixture of these, tetrastearyl titanate, potassium titanyl oxalate, lithium titanyl oxalate and the like can be listed, but it is not particularly limited. Besides, a complex oxide whose main metallic element is a titanium compound is, for example, produced from a titanium alkoxide compound and alkoxide compounds such as silicon, aluminum, zirconium, and germanium through the sol-gel process of coordination chemistry.

[0021]

Moreover, examples of bismuth compounds include bismuth alkoxide such as bismuth acetate and bismuth oxalate. Examples of aluminum compounds include aluminum butoxide,

aluminum isopropoxide and aluminum ethoxide. As examples of antimony compounds, antimony trioxide and the like can be listed.

[0022]

Other than the compounds listed in the above, examples include germanium ethoxide, zirconium ethoxide, zirconium propoxide, zirconium oxalate, hafnium butoxide, triphenyl yttrium, yttrium trifluoromethanesulfonic acid and scandium trifluoromethanesulfonic acid. Further, as examples of lanthanoid compounds, lanthanum acetate, lanthanum isopropoxide, lanthanum benzoate, cerium isopropoxide, triphenyl samarium, samarium oxalate, samarium isopropoxide, europium acetate, ytterbium acetate and the like can be listed.

[0023]

In addition, considering a reaction speed of an open ring polymerization and a hue of polyester to be obtained in the end, the amount of polymerization catalysts to be added is preferably 1 ~ 10000ppm in metal weight against cyclic polyester oligomers. Further, it is even more desirable to be 10 ~ 3000ppm in metal weight, and 20 ~ 1500ppm in metal weight is even more preferable.

[0024]

In the method to blend cyclic polyester oligomers and polyester of the present invention, a method in which blending is conducted continuously using one or more extruding machines is desirable. Examples of extruding machines include 2-axis extruding machine. Supplying methods of cyclic polyester oligomers and polyester are not particularly limited. However, a method in which cyclic polyester oligomers and polyester are supplied at the same time from a feed opening is preferable. Furthermore, polyester to be used can be supplied in a molten state, or polyester that is made into chips can be supplied. At this point, it is preferred that the particle diameter of polyester that is made into chips is as small as possible.

[0025]

Moreover, examples of methods to remove moisture that exists in polyester and cyclic polyester oligomers and a reaction solvent to be used when cyclic polyester oligomers are synthesized include, a method in which polyester and cyclic polyester oligomers are supplied to an extruding machine having a vent hole, and then under a temperature condition in that the temperature is at the crystallizing temperature of polyester or lower, the degree of pressure reduction of the vent hole is set

at 6650Pa or less, and preferably at 1330Pa or less. It is desirable because removing moisture that exists in polyester and cyclic polyester oligomers and a reaction solvent used when cyclic polyester oligomers are synthesized enables to control hydrolysis of generated polyester and degradation of hue of polymers.

[0026]

In the present invention, when a reaction time is short, a lot of unreacted materials are left. On the other hand, when a reaction time is long, polyester is thermally decomposed. As a result, hue is deteriorated or polymerization degree drops, and it is also not preferable in view of productivity. Therefore, a heating time for cyclic polyester oligomers and polyester is preferably 1 ~ 120 minutes. A heating time of 1 ~ 90 minutes is more desirable and that of 1 ~ 60 minutes is even more desirable. In the present invention, it is sufficient when a purity of cyclic polyester oligomers is 60% or more. However, preferably cyclic polyester oligomers with a purity of 70% or more are used, and using those with a purity of 80% or more is even more preferable. Further, by controlling the ratio of straight polyester oligomers contained in generated cyclic polyester oligomers at 10% or less, the amount of oligomers in polyester to be generated can be reduced. As a result, polyester with a higher degree of polymerization can be obtained and thus it is preferable. The ratio of 5% or less is more desirable and that of 2% or less is even more desirable.

[0027]

In the present invention, to improve thermal resistance and hue, prior art phosphorus compounds or cobalt compounds may be added to ultimately obtained polyester at any point. Further, in the present invention, if necessary, publicly known compounds, for example inorganic particles for frosting, antioxidant, heat insulator, antistatic agent, ultraviolet absorption agent and so forth can be added and contained.

[0028]

As described in the above, in the present invention, cyclic polyester oligomers are blended with

polyester with a weight ratio of 0.01 ~ 0.9 against polyester, then heating is conducted at a temperature in the range of 200 ~ 320 degrees Celsius. Therefore it becomes possible to obtain polyester with a high degree of polymerization more economically compared to prior art solid phase polymerization processes and open ring polymerization processes.

[0029]

[Embodiments]

Using examples in the below, the present invention is explained further in detail. Besides, physical properties in examples were measured in methods explained below.

(1) Degree of intrinsic viscosity of polymers [η]

Orthochlorophenol was used as a solvent and measurements were conducted at 25 degrees Celsius.

(2) Purity of cyclic polyester oligomers

Calculations were conducted with liquid chromatography using ratio of peak surface areas for oligomers of each polymerization degree that is identified in advance (UV absorption standard : %).

(3) Hue of polyester

Measurements were conducted using a color meter manufactured by Suga Testing Apparatus Co. (SM color computer type SM-3) and as Hunter values (L, a, b values).

[0030] Example 1

Seven kilograms of polyethylene terephthalate chips with an intrinsic viscosity of 0.80 (PET), 3 kilograms of cyclic polyethylene terephthalate powder (99% pure), and as a polymerization catalyst 534 milligrams of tetraisopropyl titanate ($Ti = 30\text{ppm}$) were supplied to a feed opening of a 2-axis extruding machine with a vent hole at a rate of 0.3kg/ minute . Rotation was conducted at a screw rotation speed of 70rpm. While reducing the pressure to 1000Pa , heating was conducted using an electric heater and the temperature was maintained at 100 degrees Celsius. A normal pressure was

restored using nitrogen, and then it was heated to 280 degrees Celsius and the screw rotation speed was adjusted so that an average residence time became 10 minutes. Subsequently, molten polymers were discharged in a gut form, and then processed through a cooling bath and cut into a pellet form with a cutter to obtain polyethylene terephthalate.

[0031]

The intrinsic viscosity of obtained polyethylene terephthalate was 1.52, the hue b-value was 5.0, and polyester with a high degree of polymerization was obtained.

[0032] Example 2

Fifty kilograms of polybutylene terephthalate with an intrinsic viscosity of 0.66 (PBT), 17.5 kilograms of cyclic polybutylene terephthalate powder (74% pure), and as a catalyst 3.14 grams of tin tetraisopropyl ($\text{Sn} = 60\text{ppm}$) were supplied to a feed opening of a 2-axis extruding machine at a rate of 0.5kg/ minute. An average residence time at the extruding machine was 5 minutes, and the reaction was conducted at 250 degrees Celsius under nitrogen. The intrinsic viscosity of obtained reaction product was 1.20, and the hue b-value was 3.9.

[0033] Examples 3, 6, 7, 10 and 11

According to the procedures of Example 1, open ring polymerizations were conducted using 2-axis extruding machines with vent and under conditions shown in Table 1. The results are shown in Table 1. In any cases polyester with a high degree of polymerization was obtained as aimed.

[0034] Example 4

Eighty kilograms of polypropylene terephthalate with an intrinsic viscosity of 0.83 (PPT), 55 kilograms of cyclic polyethylene terephthalate powder (85% pure), and as catalysts 59.9 grams of samarium isopropoxide ($\text{Sm} = 500\text{ppm}$) and 40.4 grams of zirconium propoxide ($\text{Zr} = 250\text{ppm}$) were supplied to a feed opening of a 1-axis extruding machine at a rate of 0.5kg/ minute. An average residence time at the extruding machine was 2 minutes, and the reaction was conducted at 290 degrees Celsius under nitrogen. The intrinsic viscosity of obtained polyester was 1.43, and the hue b-value was 4.1.

[0035] Examples 5, 9 and 12

According to the procedures of Example 2, open ring polymerizations were conducted with conditions shown in the table. Besides, in Example 5 and Example 12, polymers in a molten state were used, and in Example 9, polybutylene terephthalate (PBT) in a chip form was used. The results are shown in the following table. In any cases polyester with a high degree of polymerization was obtained as aimed.

[0036] Examples 8 and 13

According to the procedures of Example 4, open ring polymerizations were conducted with conditions shown in the table. Besides, in Example 8, polymers in a molten state were used, and in Example 13, polyethylene terephthalate (PET) in a chip form was used. The results are shown in the following table. In any cases polyester with a high degree of polymerization was obtained as aimed.

[0037] Comparative Example 1

A reaction was conducted in the same manner as in Example 1 except that 11 kilograms of polyethylene terephthalate with an intrinsic viscosity of 0.66 (PET) and 11 kilograms of cyclic polyethylene terephthalate (98% pure) were used, the reaction was conducted at 300 degrees Celsius for 240 minutes, and 0.78 kilograms of titanium isopropoxide ($Ti = 12000ppm$) were added as a catalyst. However, the intrinsic viscosity of obtained polymers was 0.57, and the hue b-value was 17.0.

[0038] Comparative Example 2

Three hundred grams of polyethylene terephthalate with an intrinsic viscosity of 1.65 (PET) and 150 grams of cyclic polybutylene terephthalate (65% pure) were stirred in a test tube purged with nitrogen, and 896 milligrams of tin isopropoxide ($Sn = 2000ppm$) were added as a catalyst. While stirring with stirring blade at a speed of 30rpm, it was heated to 280 degrees Celsius and the reaction was completed in 25 minutes. Consequently, it was discharged in a strand form into cold water, and then immediately cut to obtain pellets of polyester. As a result, the intrinsic viscosity of obtained polymers was 0.49, and the hue b-value was 9.8.

[0039] Comparative Example 3

A reaction was conducted in approximately the same manner as in Example 1 except that 10 kilograms of polyethylene terephthalate with an intrinsic viscosity of 0.45 (PET) and 3.5 kilograms of cyclic polypropylene terephthalate (85% pure) were used, the reaction was conducted at 290 degrees

Celsius for 15 minutes, and 4.1 grams of antimony trioxide ($Sb = 500\text{ppm}$) and 33. 7 grams of yttrium triflate ($Y = 2000\text{ppm}$) were added as catalysts. However, the intrinsic viscosity of obtained polymers

was 0.55, and the hue b-value was 11.0.

[0040] Comparative Example 4

A reaction was conducted in approximately the same manner as in Example 1 except that 10 kilograms of polybutylene terephthalate with an intrinsic viscosity of 0.66 (PBT) and 5 kilograms of cyclic polybutylene terephthalate (80% pure) were used, the reaction was conducted at 190 degrees Celsius for 120 minutes, and 7.5 grams of aluminum ethoxide ($\text{Al} = 250\text{ppm}$) were added as a catalyst. However, the intrinsic viscosity of obtained polymers was 0.53, and the hue b-value was 18.1.

[0041] Comparative Example 5

One thousand grams of polyethylene terephthalate with an intrinsic viscosity of 0.70 (PET) and 490 grams of cyclic polypropylene terephthalate (85% pure) were stirred in a test tube purged with nitrogen, and 293 milligrams of tin isopropoxide were added as a catalyst so that the tin atom content became 200ppm. While stirring with stirring blade at a speed of 30rpm, it was heated to 330 degrees Celsius and the reaction was completed in 20 minutes. Consequently, it was discharged in a strand form into cold water, and then immediately cut to obtain pellets of polyester. As a result, the intrinsic viscosity of obtained polymers was 0.60, and the hue b-value was 16.5.

[0042]

[Table 1]

	Cyclic Polyester Oligomers (A)		Polyester (B)		Weight Ratio (A) / (B)	Reaction Temperature (°C)	Reaction Time (Minutes)	Catalysts		Extruding Machine			Generated Polymers
	Oligomer Type	Purity	Polymer Type	Intrinsic Viscosity				Compound Against (A) (ppm)	2-axis with vent (Pressure Reduction Degree (pa))	1-axis	None	Intrinsic Viscosity	
Example 1	PET ¹	99	PET	0.80	0.43	280	10	Ti(0/Pr) ₄	30	O(1000)			1.52 5.0
Example 2	PBT ²	74	PBT	0.66	0.35	250	5	Sm(0/Pr) ₄	60	O			1.20 3.9
Example 3	PPT ³	80	PBT	0.56	0.49	230	25	Sb ₂ O ₃	55	O(1000)			1.37 3.7
Example 4	PET	85	PPT	0.83	0.69	290	2	Sm(0/Pr)	500/25				1.43 4.1
Example 5	PET	95	PBT	0.67	0.45	310	15	Sb ₂ O ₃ /Ti(0/Pr) ₄	70/30	O	O		1.40 5.8
Example 6	PPT	75	PET	0.74	0.50	300	70	Bi(0Ac) ₃ ⁴	1500	O(1000)			1.33 6.6
Example 7	PBT	89	PPT	0.68	0.10	295	15	Ti(0/Bu) ₄	15	O(1000)			1.45 4.5
Example 8	PPT	60	PET	0.66	0.25	280	18	Y(0iT) ₃ ⁴	30				1.01 5.1
Example 9	PBT	90	PBT	0.51	0.33	235	3	Sn(0/Pr) ₄	90	O	O		1.45 4.2
Example 10	PPT	85	PPT	0.67	0.02	255	80	Sm(0/Pr) ₃	1000	O(1000)			1.01 6.9
Example 11	PBT	85	PET	0.66	0.35	315	12	Al(0Et) ₃	2000	O(1000)			1.33 7.5
Example 12	PBT	75	PPT	0.84	0.53	245	100	GaD ₂	30	O			1.11 6.6
Example 13	PET	80	PET	0.90	0.46	280	30	Ti(0/Pr) ₄	5000				1.22 7.1
Comparative Example 1	PET	98	PET	0.66	1.00	300	240	Ti(0iPr) ₄	12000	O(1000)	O		0.57 17.0
Comparative Example 2	PBT	65	PET	1.65	0.50	280	25	Sn(0/Pr) ₃	2000		O	0.49	9.8
Comparative Example 3	PPT	85	PET	0.45	0.35	290	15	Sb ₂ O ₃ /Y(0/Ti) ₃	500/2000	O(1000)			0.55 11.0
Comparative Example 4	PBT	80	PBT	0.66	0.50	190	120	Al(DEt) ₃	250	O(1000)			0.53 18.3
Comparative Example 5	PPT	85	PET	0.70	0.49	130	20	Sn(0/Pr) ₄	200		O	0.60	16.5

¹:PET = Polyethylene terephthalate
Bi (OAc) O₃ = Bismuth acetate²:PBT = Polybutylene terephthalate
Y(Ot)₃ = Yttrium Triflate³:

PPT = Polypropylene terephthalate

[0043]

[Advantages of the Invention]

With the production method of polyester with a high degree of polymerization of the present invention, it becomes possible to obtain polyester with a high degree of polymerization in a shorter time period compared to prior art production methods.

Continued to the front page

F term (reference)	4J029	AA03	AB05	AB04	AC02	AC04
		AC05	AD01	BA03	BA04	BA05
		BA08	BD06A	CB06A	CC06A	
		CG09X	JA061	JB131	JB171	
		JC021	JC121	JC751	JF221	
		JF261	JF271	JF321	JF331	
		JF341	JF361	JF371	JF471	
		JF481	KB03	KB05	KB15	
		KC02	KD01	KD02	KE05	LA10
		LA20				